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B1C
B1V
Selected US specifications from IPC sub-class B01F

(54) Emusifying equipment and method

(57) Emulsifying equipment comprises two cylindrical members (21, 29) between which an annular gap (11b) is formed. Liquids to be mixed are fed (via 12a, 12b) into the gap while relative rotation is maintained between the two cylindrical surfaces defining the gap (i.e. inner member 29 is rotated via shaft 30). The liquids in the gap are subjected to a substantially uniform shear in the range 3000 to 40,000 mm/sec.

During mixing the liquids can flow through two gaps (11b, 11a) in passing from the inlet (12a, 12b) to the outlet (13). Each liquid is separately pumped into the gap since the rotating cylindrical surface(s) has no significant pumping action.

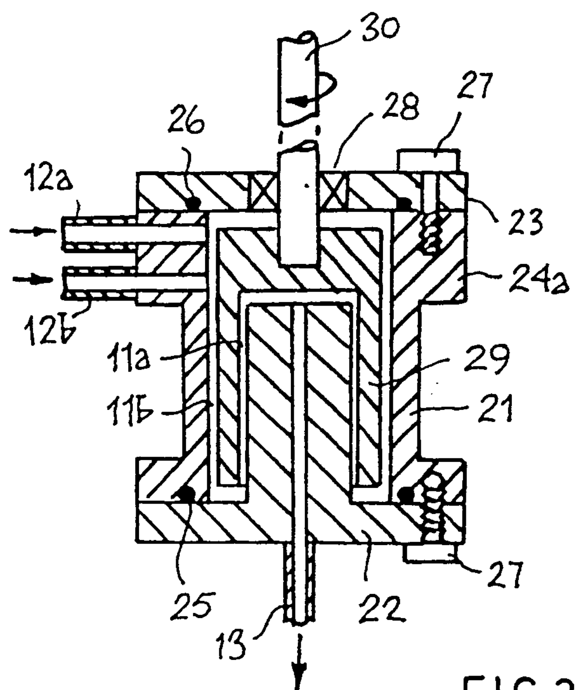

FIG.2

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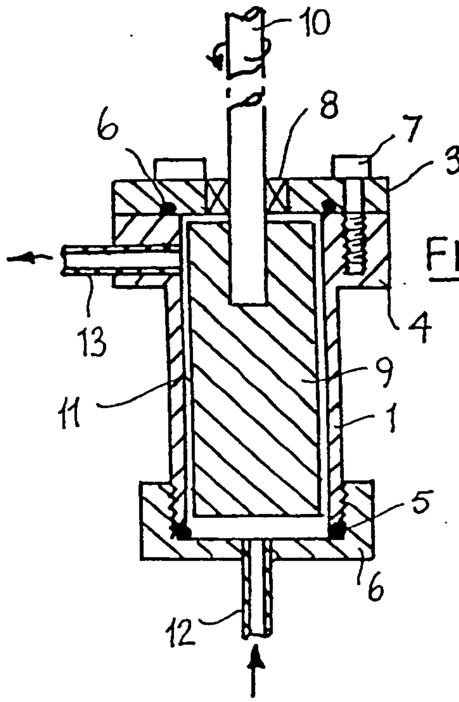


FIG.1

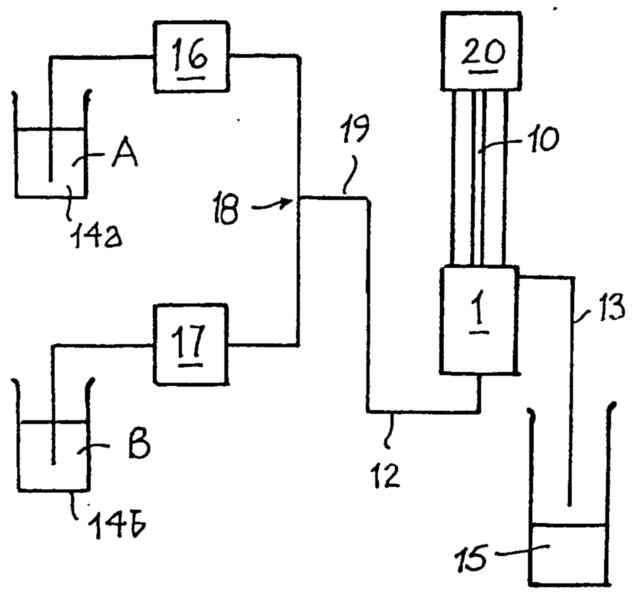


FIG.3

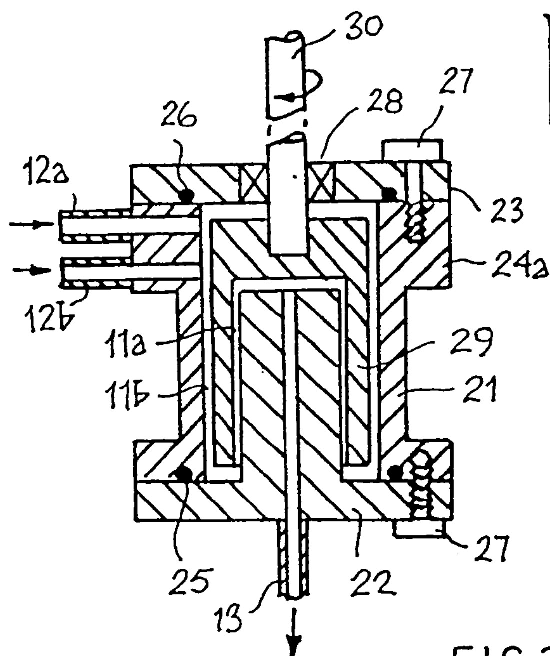
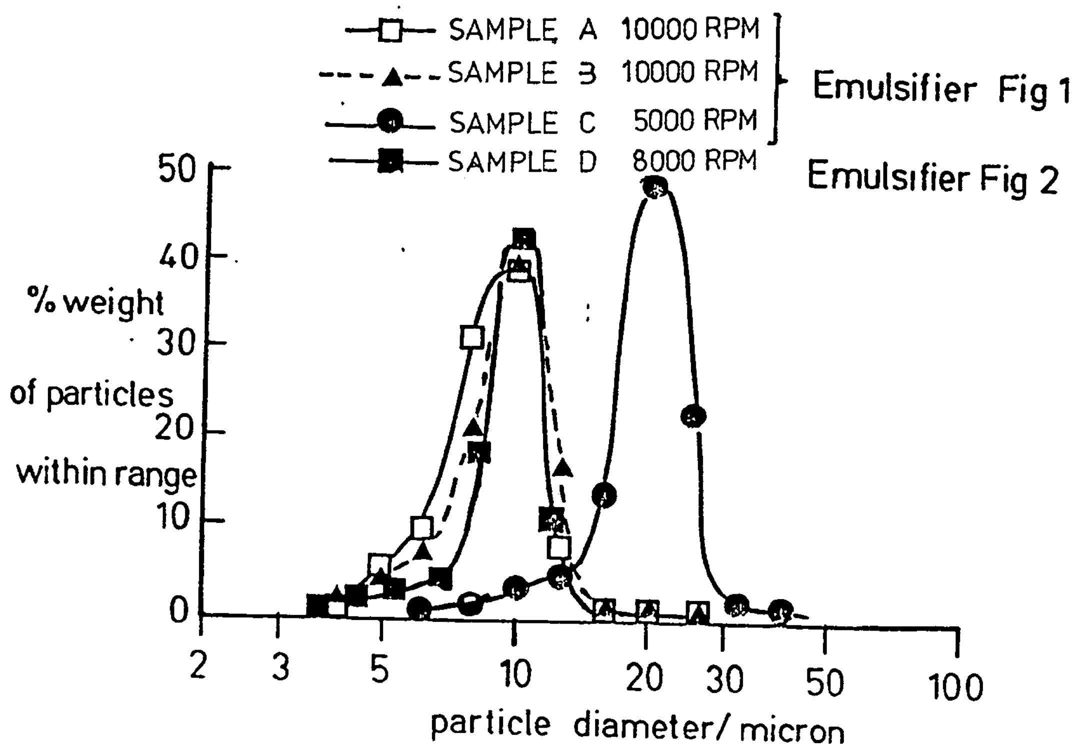
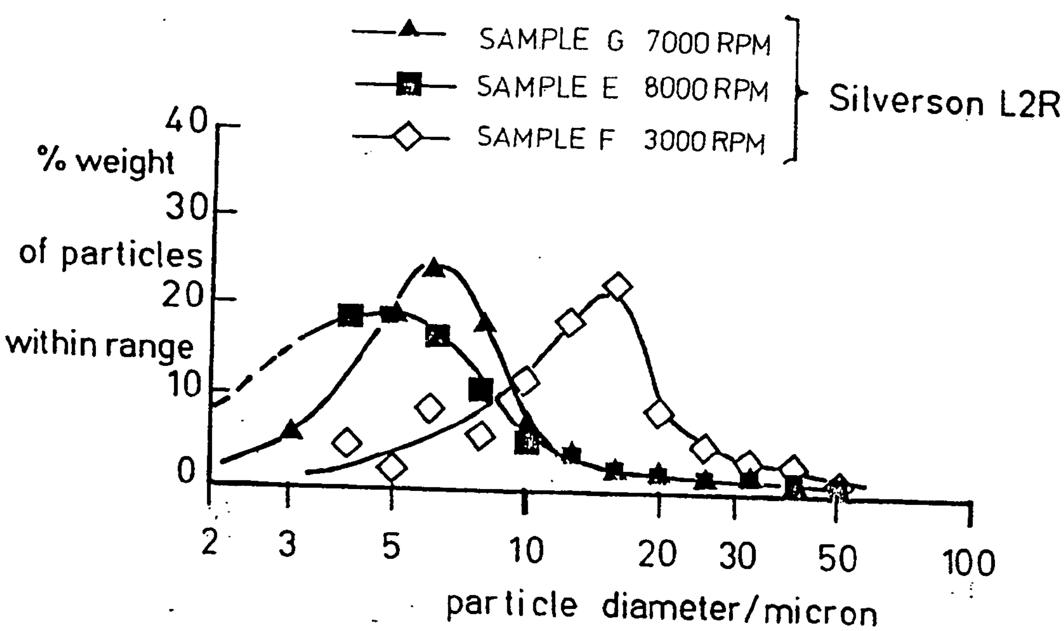


FIG.2


SAMPLE A 10000 RPM
SAMPLE B 10000 RPM
SAMPLE C 5000 RPM
SAMPLE D 8000 RPM
Emulsifier Fig 1
Emulsifier Fig 2
% weight of particles within range
0
10
20
30
40
50
2
3
5
10
20
30
50
100
particle diameter/micron


SAMPLE G 7000 RPM
SAMPLE E 8000 RPM
SAMPLE F 3000 RPM
Silverson L2R
% weight of particles within range
0
10
20
30
40
2
3
5
10
20
30
50
100
particle diameter/micron

SPECIFICATION

Improvements in mixing

This invention relates to an improved method of and apparatus for mixing two or more liquid streams. The invention has particular utility in the production of an emulsion.

In many applications it is desirable to create an emulsion in which the particles of the disperse phase are as uniform as possible in size. The majority of emulsifiers currently available produce a wide range of particle sizes. This is largely because they operate under conditions of extreme turbulence at the point where the emulsion is formed usually by means of a stirrer rotating at high speed within a cage.

For any emulsion, it is possible to define a range of diameters of the particles of the disperse phase from Dmin to N×Dmin such that the particles within this range make up 95% of the total weight of the particles of the disperse phase. With a typical prior art emulsifier the value of N for any emulsion produced will typically be greater than eight.

The present invention seeks to produce an emulsion in which N is of the order of no more than 3 and in which there is a relatively sharp cut-off at the large particle size end of the range.

According to one aspect of the invention a method of mixing two or more liquid streams comprises forcing the streams to flow together through a mixing region in which a substantially uniform shear is maintained.

Suitably the shear is in the range 3000 mm/sec to 40,000 mm/sec.

Conveniently the shear gradient in the mixing region is in the range 3000 sec^{-1} to 40,000 sec^{-1}. Desirably the rate at which the streams are forced into the mixing region is controlled independently of the shear generated in the mixing region. To achieve independence between flow rate and shear, the shear must be generated by an arrangement which does not significantly pump the fluids and one structure which achieves this provides the mixing region in a narrow annular gap formed between circular cylindrical members between which relative rotation is maintained.

In one embodiment of the invention the outer cylindrical member is stationary and a rapidly rotating drum defines the inner cylindrical member.

In a second embodiment of the invention the inner cylindrical member remains stationary while the outer member rotates.

In a third embodiment of the invention a cylindrical shell closed at one end rotates between two fixed cylindrical members the axes of all three cylindrical members being coincident thereby defining two connecting annular regions each providing uniform shear but not necessarily the same shear.

Desirably turbulence in the liquid streams entering the gap, leaving the gap, and during transport through the gap is minimised.

According to a further aspect of the invention, mixing apparatus comprises inner and outer circular cylindrical surfaces between which an annular gap is formed, means to rotate one of the cylindrical surfaces, relative to the other, about the axis of the gap, means to convey at least two streams of liquid to be mixed to one end of the gap and means to remove a stream of mixed liquid from the other end of the gap.

In one embodiment of apparatus, the gap has an axial length which is at least thirty times and preferably at least fifty times the width of the gap and diameter of the inner member is at least ten times, preferably at least twenty times, the width of the gap. Rotational speeds in the region of 5000 rpm between the surfaces defining the gap are expected to be typical during a mixing operation.

An emulsion in which 95% of the weight of the particles of the disperse phase can be made up from particles having diameters lying within the range of Dmin to 3×Dmin where Dmin is the minimum diameter of the particles within this fraction, represents a still further aspect of the invention.

The invention will now be further described, by way of example, with reference to the accompanying drawings, in which:

Figure 1 shows a first embodiment of emulsifier in accordance with the invention in axial cross-section,

Figure 2 shows a second embodiment of emulsifier in accordance with the invention also in axial cross-section,

Figure 3 shows the general layout of the emulsifier of either Fig. 1 or Fig. 2 and its ancillary liquid stream supply means and emulsion collecting means, and

Figures 4 and 5 show respectively, typical graphs of the size distribution of the disperse phase particles produced in the emulsifiers of Figs. 1 and 2, and the disperse phase particles produced in a prior art mixing unit.

The emulsifier shown in Fig. 1 comprises a circular cylindrical cage 1 (e.g. of metal) closed at its lower end by a bottom cap 2 and closed at its upper end by a top cap 3 clamped to a

flange 4 of the cage 1. Annular seals 5 and 6 are provided to render the cage 1 and its caps 2, 3 leak-tight against the liquid pressures generated during use of the emulsifier. Bolts 7 secure the top cap 3 in place.

Rotatably supported in the cage 1 from a bearing 8, is a circular cylindrical rotor 9 fast on a drive shaft 10. Between the outer cylindrical surface of the rotor 9 and the inner cylindrical surface of the cage 1 is a narrow annular gap 11 which provides a mixing chamber in which the emulsification of two liquid streams occurs. The liquids to be emulsified are fed together into the gap 11 via an inlet 12 in the bottom cap 2 and leave the gap 11 via an outlet 13 in the flange 4. Alternatively the flow direction can be reversed and the liquids to be emulsified may be fed into the gap 11 via 13 and leave the gap via 12.

The emulsifier shown in Fig. 2 comprises a circular cylindrical cage 21 (e.g. of metal) closed at its lower end by a cap-and-plug unit 22 and closed at its upper end by a top cap unit 23 clamped to a flange 24a of the cage 21. The cap-and-plug unit 22 is likewise clamped to a flange 24b of the cage 21. Annular seals 25 and 26 are provided to render cage 21 and its cap units 22, 23 leak-tight against liquid pressure generated during use of the emulsifier. Bolts 27 secure the units 22 and 23 in place.

Rotatably supported in the cage 21 from a bearing 28 is an annular cylindrical rotor 29 closed at the top end and fast on a drive shaft 30. Between the cylindrical inner surface of the rotor 29 and the outer cylindrical surface of the lower cap-and-plug unit 22 is a narrow annular gap 11a which provides a final mixing zone in which the final emulsification is completed. Between the outer cylindrical surface of the rotor 29 and the inner cylindrical surface of the cage 21 is a second annular gap 11b which is preferably wider than the gap 11a and which provides for preliminary mixing and emulsification. The liquids to be emulsified are fed into the gaps 11a and 11b via inlets 12a and 12b (it is envisaged that one of the liquids may be added through 12a and the other through 12b or alternatively that both liquids are fed through 12a) and leave via the outlet 13 which is provided centrally in the cap-and-plug unit 22.

Fig. 3 shows the connection of the emulsifier of Fig. 1 with its ancillary equipment. The ancillary equipment consists of a reservoir 14a for a liquid A, a reservoir 14b for a liquid B and respective pumps 16, 17 for feeding the liquids A and B at a controlled rate and in the desired proportion to a mixing tee 18. The inlet 12 is connected to the tee 18 by a duct 19. The motor for rotating the shaft 10 is shown at 20, and a collecting vessel for the emulsion is shown at 15.

When using the emulsifier shown in Fig. 2, the tee 18 may be ommited and the two liquids fed directly to the two inlets 12a and 12b, or alternatively if only one of the inlets is used, then this takes the place of inlet 12 of Fig. 3.

Should a third or further additional stream(s) be required this/these can be fed to the gap 11 from one or more further reservoirs (not shown) using a further pump (also not shown) for each. Where three or more streams are needed, the tee 18 can act as the initial contact point for all the streams or one or more further mixing tees can be provided downstream of the tee 18.

The principle of operation of the emulsifiers of Figs. 1 and 2 is that each forms the emulsion under conditions of substantially uniform shearing velocity. In the case of the emulsifier of Fig. 1 this is achieved by feeding the two phases (A and B) which are to form the emulsion through the inlet 12 into the gap 11 between the cylindrical cage 1 and the cylindrical rotor 9 while the latter rotates at a high speed. In one device constructed according to Fig. 1 and tested, the rotor 9 was 60mm in length and 26.5mm in diameter. The gap 11 between the rotor 9 and the wall of the cage 1 was 1mm wide. The motor 20 was from a Silverson heavy duty laboratory mixer and could rotate the rotor 9 at speeds between 3000 and 12000 rpm, thereby achieving shearing rates of between 4000 sec^{-1} and 16000 sec^{-1} on the assumption of laminar flow.

In the case of the emulsifier of Fig. 2, the two phases are fed separately to the gap 11b at constant flow rates through the inlets 12a and 12b. The phase which acts as the better lubricant for the bearing 28 can be introduced at 12a, the upper of the two inlets. In one device constructed according to Fig. 2, and tested, the rotor 29 was a cylindrical shell whose length was 66 mm, whose outer diameter was 28.6 mm, and whose inner diameter was 21mm. The annular gap 11b between the rotor and the inner surface of the cage 21 was 1 mm, and the gap 11a between the inner surface of the rotor 29 and the outer surface of the cap and plug unit 22 was 0.5 mm. Rotor speeds of 3000 and 12000 rpm would then give shearing rates within the inner annulus of about 6000 sec^{-1} and 24000 sec^{-1} respectively on the assumption of laminar flow. The embodiment shown in Fig. 2 is designed so as to make a primary emulsion in the outer and wider annular gap 11b; the primary emulsion then passing into the narrow inner annular gap 11a where the larger particles of the disperse phase formed in the gap 11b are further broken down. It is expected that the apparatus of Fig. 2 will provide an emulsion with more uniform particles than that produced in the apparatus of Fig. 1.

Whereas most prior art emulsifiers act as their own pump so that liquid circulates vigorously through the emulsification region, the rotation of the rotor 9 or 29 in the devices of Fig. 1 and 2 generates little or no pumping action. For this reason the two liquids A and B, which are to

form the emulsion, are separately forced from their reservoirs 14a and 14b to the emulsifier chamber 11 (11a, 11b).

The sharp flow deflections induced in the streams as they enter the emulsion chamber at 12 (12a, 12b) and leave the emulsification chamber at 13, as well as the deflections induced when the flow of liquid changes direction at the corners of the rotor 9 or 29, may induce undesirable turbulence and cause formation of very small particles of the disperse phase. This effect can be reduced by suitable shaping and smoothing of the relevant parts.

The invention is further illustrated by the following Examples:–

Example 1

In typical experiments using the embodiment of the invention shown in Fig. 1, 100–120°C grade petroleum containing 1 to 3% w/w of a non-ionic surface active agent Span 80 as an emulsifier was placed as liquid A in reservoir 14a and water containing 30% w/w of colloidal silicon dioxide from which it was eventually desired to make spherical solid particles was placed as liquid B in reservoir 14b. Liquids A and B were drawn at rates of 12ml/min each through tee 18 to inlet 12. An Orlita piston pump was used at 16 to pump the petroleum phase and a Watson Marlow peristaltic pump was used at 17 to pump the aqeuous phase. Under these conditions, a water-in-oil emulsion was formed in the gap 11 which contained equal volumes of the dispersion medium (petroleum) and disperse phase (aqueous). With rotor speeds of respectively 10,000 and 5,000 rpm, particles of the disperse phase, after hardening, separation and drying had maximum diameters of about 10 and 20 microns, respectively. It is possible that the particles of the disperse phase before drying were somewhat larger than this since it is well known that silica gel particles contract significantly on drying. The shearing rates applied in this example, assuming laminar flow, were 28,000 and 14,000 sec^{-1}, respectively.

An emulsifier in accordance with this invention is expected to be useful for producing emulsions containing particles up to about 100 microns in diameter. In order to produce particles larger than those of Example 1, the width of the gap 11 can be increased and the rotation rate of the rotor 9 can be reduced.

Fig. 4 shows the particle size distributions measured by a Coulter Particle Size Analyser for three samples (Samples A, B and C) produced in the apparatus of Fig. 1. The data are presented as plots of the percentage of the total weight of the particles which are to be found in successive diameter ranges where the ratio of the maximum diameter to the minimum diameter in any range is approximately 1.25. The three separate experiments carried out gave the following means particle diameters and standard deviations when calculated on a weight fraction basis:

Sample code with rotor speed in rpm in brackets	Mean Particle Diameter in Microns	Standard deviation about the means as a multiplier of the mean diameter
A (10,000)	8.7	1.30
B (10,000)	9.3	1.40
C (5,000)	19.5	1.35

It may be noted from Fig. 4 that the cut-off at the upper end of the particle size diameter range is sharp while there is a "tail" at the lower end. It is thought that this tail may be due to failure to control areas of turbulent flow at the inlet and outlet of the emulsifier.

Example 2

In a typical experiment carried out using the embodiment of the invention shown in Fig. 2, 100–120°C grade petroleum containing 1–3% w/w of a non-ionic surface active agent Span 80 as an emulsifier was placed as liquid A in reservoir 14a and water containing 30% w/w of colloidal silicon dioxide was placed as liquid B in reservoir 14b. Liquid A was fed at 12ml/min through the inlet 12a and liquid B was fed at 12ml/min through inlet 12b. In this way the petroleum acted as a lubricant for the bearing 28, and contact of the bearing with the abrasive silica sol was minimised. An Orlita piston pump was used at 16 to feed the petroleum phase and a Watson-Marlow peristaltic pump was used at 17 to feed the aqueous silica sol. A water-in-oil emulsion was formed in the gap 11b and passed, through the action of the pumps, into gap 11a, where the larger particles of emulsion were further broken up. At a rotor speed 8000 rpm the maximum shearing rate in the gap 11a, assuming laminar flow, was about 16,000 sec^{-1}.

Fig. 4 shows the particle size distribution measured by a Coulter Particle Size Analyser for a sample (Sample D) produced in the apparatus of Fig. 2. The mean particle diameter and the standard deviation of the particle diameter range calculated on a weight fraction basis were:

Sample code with rotor speed in rpm in brackets	Mean Particle Diameter in microns	Standard deviation about the mean as a multiplier of the mean diameter
D (8000)	10.0	1.32

It may be noted that, apart from the tails to lower and higer particle diameters, the main peak in the particle size distributions in Example 2 is narrower than those obtained in Example 1. This confirms that a double annular gap gives a particle size uniformity which is superior to that obtained with a single annular gap.

Example 3
Fig. 5 shows the particle size distributions obtained for samples emulsified using a prior art emulsifier namely a Silverson heavy duty laboratory mixer. The data are presented in the same way as those in Fig. 4. Three separate experiments carried out gave the following means particle diameters and standard deviations:

Sample code with rotor speed in rpm in brackets	Mean Particle Diameter in microns	Standard deviation about the mean as a multiplier of the mean diameter
E (8000)	7.3	1.90
F (3000)	12.7	1.72
G (7000)	7.0	1.97

The particle size distribution obtained with a state of the art emulsifier unit is substantially wider than that obtained using the emulsifier units of the invention.

With the equipment in accordance with the present invention it will be seen that emulsions can readily be prepared in which 95% of the weight of the particles of the disperse phase is made up from particles having diameters falling within a threefold range, where the diameter range is defined as the ratio of the maximum diameter to the minimum diameter of the particles falling within the range (four standard deviations). By contrast a prior art emulsifier gave emulsions in which the diameter range required to contain 95% of the weight of the particles of the disperse phase was around ten.

Thus preferred embodiments in accordance with the invention achieve a very significant improvement in the particle size uniformity of an emulsion.

CLAIMS

1. A method of mixing two or more liquid streams which comprises forcing the streams to flow together through a mixing region in which a substantially uniform shear is maintained.

2. A method as claimed in claim 1, in which the shear is in the range 3000 mm/sec to 40,000 mm/sec.

3. A method as claimed in claim 1, in which the shear gradient in the mixing region is in the range 3000 sec^{-1} to 40,000 sec^{-1}.

4. A method as claimed in any one preceding claim, in which the rate at which the streams are forced into the mixing region is controlled independently of the shear generated in the mixing region.

5. A method as claimed in claim 4, in which the mixing region is provided in the annular gap between two circular cylindrical member between which relative rotation is maintained.

6. A method as claimed in claim 5, in which the outer cylindrical member is stationary and a rapidly rotating drum defines the inner cylindrical member.

7. A method as claimed in claim 5, in which the inner cylindrical member remains stationary while the outer member rotates.

8. A method as claimed in claim 5, in which a cylindrical shell closed at one end rotates between two fixed cylindrical members, the axes of all three cylindrical members being coincident thereby defining two connecting annular regions.

9. A method as claimed in any one of the preceding claims, in which turbulence in the liquid streams entering the gap, leaving the gap, and during transport through the gap is avoided.

10. Mixing apparatus comprising inner and outer circular cylindrical surfaces between which an annular gap is formed, means to rotate one of the cylindrical surfaces, relative to the other, about the axis of the gap, means to convey at least two streams of liquid to be mixed to one end of the gap and means to removed a stream of mixed liquid from the other end of the gap.

11. Mixing apparatus as claimed in claim 10, in which the gap has an axial length which is at least thirty times the width of the gap.

12. Mixing apparatus as claimed in claim 10, in which the gap has an axial length which is at least fifty times the width of the gap.

13. Mixing apparatus as claimed in any one of claims 10 to 12, in which the diameter of the inner member is at least ten times the width of the gap.

14. Mixing apparatus as claimed in any one of claims 10 to 12, in which the diameter of the inner member is at least twenty times the width of the gap.

15. Mixing apparatus as claimed in any one of claims 10 to 14, in which the means to rotate one of the cylindrical surfaces relative to the other is capable of rotating said one surface at a rotational speed of 5000 rpm.

16. Mixing apparatus as claimed in any one of claims 10 to 15, in which a separate pump is provided for each of the streams of liquid to be mixed.

17. A method of making an emulsion substantially as hereinbefore described with reference to the accompanying drawings.

18. A method of making an emulsion substantially as hereinbefore set out in Example 1 or Example 2 hereof.

19. Mixing apparatus substantially as hereinbefore described with reference to, and as illustrated in Figs. 1 to 3 of the accompanying drawings.

20. An emulsion in which 95% of the weight of the particles in the disperse phase is made up from particles having diameters lying in the range of Dmin to 3×Dmin where Dmin is the minimum diameter of the particles within this fraction.

21. An emulsion as claimed in claim 20, in which the standard deviation about the means diameter of the particles as a multiplier of the mean diameter is less than 1.40.

22. A emulsion produced by the method of claim 1 having a characteristic substantially as shown in any one of the graphs shown in Fig. 4 of the accompanying drawings.

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